

Noise Measurement Setup for Quartz Crystal Microbalance

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Abstract. Quartz crystal microbalance (QCM) is a high sensitive chemical sensor which has found widespread spectrum of applications. There are several mechanisms that are related to fluctuation phenomena. Since the aim of our research is oriented to study the sensitivity and influence of different kind of noises on sensor resolution, we modified an existing method to measure the small frequency fluctuation of QCM. The paper describes our measurement setup, in which a quartz crystal oscillator with coated active layers and a reference quartz oscillator are driven by two oscillator circuits. Each one regulates a frequency of a crystal at the minimum impedance which corresponds to the series resonance. A data-acquisition card triggers on the rise-edges of the output signal and stores these corresponding times on which the instantaneous frequency is estimated by own-written software. In comparison to other measurement setups, our approach can acquire immediate change of QCM frequency, thus, chemical processes can be even described on the basis of high-order statistics. The experiments were provided on quartz crystals with the sorption layer of polypyrrole, which is suitable for the construction of QCM humidity sensors.

Keywords

Quartz crystal microbalance, noise measurement setup, fluctuation.

1. Introduction

The detection of chemical substances dissolved in a gas or liquid strongly requires chemical sensors which are able to adsorb reversibly the molecules of analyzed matter in amounts that depend on its concentration and on the specificity of adsorbing sites in a sensor. For the transformation of non-electric input quantity to electric signal, the sensors utilize various physical or chemical phenomena. One of universal transfer mechanisms is the change of the resonant frequency of quartz crystal.

Quartz crystal microbalance (QCM) represents highly sensitive sensor for a detection of chemical substances. QCM-based gas sensors are widely utilized as a result of their robust nature, availability and affordable interface electronics [1]. There are several mechanisms that are related to fluctuation phenomena. Generally, users require minimizing their impact on measurements in order to achieve maximal signal-to-noise ratio. However, fluctuation mechanisms can also give useful pieces of information about physical or chemical processes occurred in a sensor.

Noise measurement and its evaluation represent a new approach of extracting more selective response from resistive [2] and surface acoustic wave [3] sensors. Experimental results demonstrated that the noise spectral density of sensor's resistance fluctuations is modified by exposure to different gases as well as by exposure to different concentration of gases. Smulko et al. [4] showed that sensibility and selectivity of gas detection in Taguchi gas sensors can be increased by observing resistance noise of sensors. The observed fluctuations give more information than a single DC resistance value and therefore lead to a reduction of the number of gas sensors necessary for detection of different gas mixtures. Gomri et al [5] proposed a model of adsorption-desorption noise in metal oxide gas sensors, based on the free electron density fluctuation produced by the gas adsorption. Using this model for simulating the oxygen chemisorption – induced noise, they found that the contribution of oxygen adsorption-desorption noise to the noise spectra is a Lorentzian component having a corner frequency and low frequency magnitude which are specifics of the adsorbed gas. Thus, noise spectroscopy might be highly useful for improving gas sensors selectivity and sensitivity.

Our preliminary experimental results revealed [6], [7] that absorption of detected matter affects fluctuations in QCM. Hence we proposed a new method based on noise measurements for evaluation of QCM measurements besides standard techniques [1], [8-10]. The paper describes our measurement setup which was designed to study small frequent fluctuation of QCM.

2. Quartz Crystal Microbalance

Originally employed for mass sensing in gas-phase deposition processes, QCM has evolved into a versatile platform for both liquid and gas-phase chemical sensing. Fig. 1 illustrates its sensing principle. The core of QCM sensor is the AT-cut quartz crystal that oscillates at a resonant frequency which is determined by mass and geometry of the crystal mass as well as several other factors (temperature, applied voltage etc.). The section for the cut is chosen specially for the shear oscillations to possess the first order zero temperature coefficient which is necessary to provide thermal stability to devices.

The quartz electrodes are covered by sorption layers with affinity to the molecules of the detected matter. Sorbed matter (molecules of the detected mass) represents mass increment and the change of the viscoelastic properties of the layer which leads to the change of resonant frequency.

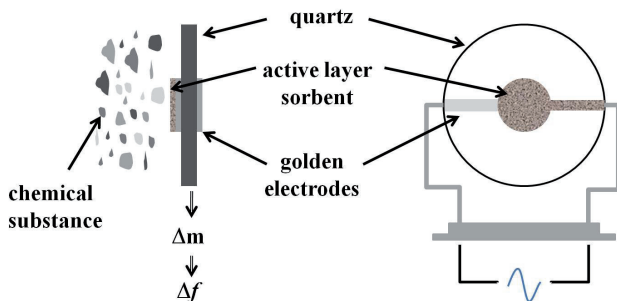


Fig. 1. QCM principle: molecules of the detected mass represent mass increment and the change of the viscoelastic properties of the layer, which leads to the change of resonant frequency.

Selectivity and sensitivity of a sensor are given by the choice of the material of the sorption layer. At a resonance frequency of 10-15 MHz, the sensitivity may reach 1 ng/cm² which corresponds to the monolayer of adsorbed substance. The mass sensitivity of QCM chemical sensors originates from Sauerbrey's equation [1]

$$\Delta f = -2f_0^2 \Delta m / A\sqrt{\mu\rho} \quad (1)$$

where f_0 is the resonant frequency of the fundamental mode of a quartz crystal, A is the area of the gold disk coated onto the crystal, ρ denotes the density of quartz crystal ($\rho = 2684 \text{ kg m}^{-3}$), and μ is shear modulus of quartz ($\mu = 2.95 \times 10^{10} \text{ Pa}$). Only mass increment of adsorbed molecules is concerned since there is no need to determine the absolute mass of the resonator. For example, a net change of 1 Hz (a crystal of fundamental frequency $f_0 = 10 \text{ MHz}$ and area $A = 19.6 \text{ mm}^2$) corresponds to the absorbed mass Δm

$$\Delta m = 8.58 \text{ ng}. \quad (2)$$

Sensor parameters (such as sensitivity, selectivity, dynamics and stability) rely not only on composition but even on deposition technique of the sorbent layer. For analytical purposes, it is imperative to find the ways of

getting the QCM sensor to respond only to what we are interested in (i.e. build sensitivity into the device).

3. Measurement Setup

There are basically three standard approaches of QCM measurement [8]: excitation impulse, network analysis and oscillators. The first approach considers that the quartz crystal after excitation with an ideal impulse will carry out damped oscillations which are determined by crystal properties. Determination of the sensor transfer function, unperturbed by the interface electronics, is a major advantage of impulse excitation as well direct access to resonant frequency and acoustic energy dissipation [10]. But in practice, this approach can be used only in limited range for precise frequency and damping measurement.

Network analysis determines the frequency shift and the shift in bandwidth (proportional to dissipation) by measuring the complex admittance around a resonance frequency and fitting resonance curves to the admittance spectra [8-10]. Due to passive operation of the quartz, it is possible to minimize parasitic influences respectively to exclude their effects by calibration, which is a remarkable advantage of this method. The essential drawback of network analysis is its high costs and large dimensions of the equipment which is to satisfy the requirements of universal application [8].

In last one, quartz crystal is a part of wideband oscillator circuit whose frequency is controlled by the crystal properties [1] and is measured by a frequency counter. The application of oscillator circuits as sensor interfaces for QCM is the most common method, since quartz as a resonant element can be excited to stable oscillation by simply structured circuits, which deliver a frequency analog output signal for easy processing in digital systems [8]. Some researchers [12] use a reference oscillator to avoid ambient effects. In that case, a counter measures the frequency difference between the output signal of the sensor based oscillator, and the output signal of the reference quartz based oscillator. In order to study fluctuation mechanisms in QCM, we modified active method to measure instantaneous frequency of QCM.

3.1 The Concept of Measurement System

In a classical measurement system, the frequency of measured oscillator is monitored by standard frequency counter whose stability is at least 10 times higher. This solution has disadvantage in additive error influences of oscillator itself, e.g. temperature of tested medium and amplifier phase instability caused by temperature fluctuations and stability of power supply voltage. Therefore, differential measurement system with two oscillators (a quartz crystal with sorbent layer and a reference quartz crystal) represents better solution.

The parasite influences (temperature, power supply) take effect on both oscillators of same circuitry and same active components; hence, they can be compensated by subtraction in frequency mixer. Thus, frequent difference between the oscillators represents only concentration change of detected matter denotes, and is given by signal mixing of both oscillators and by passage through suitable low-pass filter. Further advantage is significant reduction requirements on relative measurement accuracy of frequent difference due to decrease of its mean value. Fundamental resolution remains the same. However, the issue of this solution is frequency fluctuations of oscillators which cannot be eliminated by this differential circuitry, since frequent fluctuations of both oscillators are uncorrelated.

3.2 Choice of Oscillator Circuitry

Design of suitable interface circuits for QCM sensors requires special circuit configurations which are not to be obtained by simply modifying standard applications [8-10], [13], [14]. In order to design suitable circuit, the fundamental equivalent circuit (see Fig. 2) of piezoelectric resonator has to be considered.

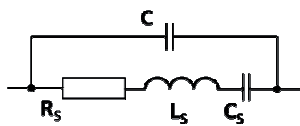


Fig. 2. Electrical equivalent circuit for quartz crystal.

The properties of quartz crystal close to a resonance frequency can be expressed by parameters of equivalent circuit, where R_s is dynamic resistance (friction damping of quartz slice, acoustic damping, acoustic damping of ambient), L_s is dynamic inductance (oscillating mass of the quartz), C_s is dynamic capacitance (elasticity of the oscillating body), C is static parallel capacitance (capacitance between quartz electrodes). Mechanical properties of quartz are represented by the components of a series equivalent circuit (R_s, L_s, C_s). With the static parallel capacitance C as the only electrical parameter, the mentioned components determine the basic frequency of an oscillating circuit. Considering Fig. 2, a series resonance frequency f_s and a parallel resonance f_p are given by

$$f_s = \frac{1}{2\pi\sqrt{L_s C_s}}, \quad f_p = \frac{1}{2\pi} \sqrt{\frac{C_s + C}{L_s C_s C}}. \quad (3)$$

Serial resonance is determined only by mechanical properties of quartz crystal whereas parallel resonance is particularly influenced by parasite capacity of crystal output pins and by all capacities, which are connected to output and caused frequency shift from serial resonance to parallel resonance. Obviously, the frequency of parallel resonance has higher instability caused by external parasite influences than frequency of serial resonance. The instability rate can be determined by ratio of serial and parallel resonance.

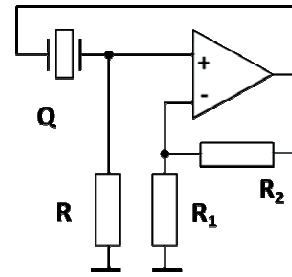


Fig. 3. Quasi-linear model of quartz crystal oscillator with serial resonance.

Newer developments of quartz oscillators resulted from steadily increasing requirements to frequency stability in measurement technique (frequency and time measurement) as well as in communications engineering, where operation in vacuum or protective gas dominates. Several circuits are commonly used for design of crystal oscillators, such as crystal oscillators with parallel resonance, crystal oscillators with serial resonance and Colpits oscillators. The frequency of Colpits type is in the range between parallel and serial resonance of quartz crystal. The $\pm 20\%$ change of parallel capacity C represents relative change 2×10^{-5} for parallel resonance and 4×10^{-9} for serial resonance. For Colpits oscillator, relative change is in the interval between these values.

Considering the values of relative change, serial resonance oscillator represents circuitry of the highest possible stability of working frequency [8-10], [13], [14]. Other solutions give significantly worse results. Fig. 3 shows fundamental quasi-linear model. Quartz crystal Q and resistor R forms band-pass with serial resonant circuitry and zero-phase shift. To satisfy the phase condition for oscillation, the feedback circuitry of oscillator is formed by non-inverting amplifier of suitable amplification whose value meets the amplitude condition for oscillation.

The frequency instability of quartz crystal oscillator is affected by instabilities of the components in quasi-linear model and, further, by instabilities associated with the nonlinear solution of amplitude condition for the oscillation. The instability of amplifier phase-transfer function represents the highest influence and its effect decreases with an increasing value of transition frequency (GBW). Considering our case, it is necessary to use an amplifier with a transition frequency of 500 MHz. Thus, the effect is reduced to the level of quartz crystal itself. Furthermore, it appears that in this case the instability of frequency is three orders lower in the magnitude than for the oscillator with parallel resonance and at least an order lower in the magnitude than for Colpits circuitry.

Also, a solution of the amplitude condition plays key role in the frequency instability. Two approaches exist. In the first approach, the amplitude of output signal is nonlinearly stabilized by the saturation of amplifier. However, the amplifier gets into a certain mode for a time, where its parameters are not guaranteed and stable. Also, influences of power instability and local thermal instability increases in the amplifier. Further, a relative high power is

on a quartz crystal itself and increasing its thermal instability. The second approach uses inertial stabilization of the amplitude, which ensures the linear mode operation of the amplifier and the limiting power on a quartz crystal to the manufacturer's guaranteed level.

3.3 Resulting Circuitry

The circuitry of each oscillator in our case uses two amplifiers. The first one is in the feedback loop and has a relatively low supply voltage due to the requested wide-transit frequency. The second one serves to amplify the stabilized low-amplitude output from the first amplifier and does not need to satisfy the high demand on phase characteristics. The output is led also to full-wave rectified LED, whose light controls the size of photo resistor. This optoisolator determines the amplification of the first amplifier and inertially stabilizes the amplitude of oscillator itself to obtain corresponding voltage, which excites LED, on the output of the second amplifier.

The signals from both oscillators are summed by an amplifier and a diode circuit reduces one half of the output signal. The results are mixing products including the differential components corresponding to the beat of two nearby frequencies. This low-frequency signal is separated by a low-pass filter whose cut-off frequency lies significantly lower than frequencies of the oscillators. This filter sufficiently suppresses any high-frequency components to avoid parasitic components in the resulting signal.

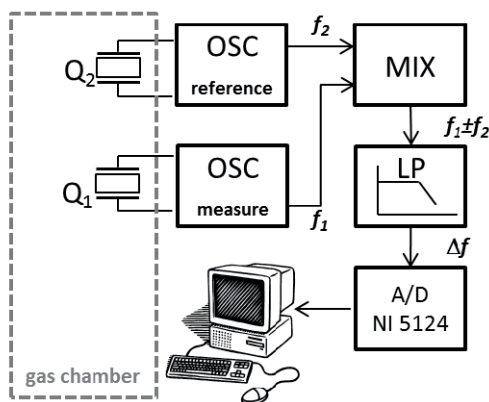


Fig. 4. Our noise measurement setup for QCM.

3.4 Evaluation

For practical solution of measurement setup, the automation and computer control of measurement have to be managed. Three methods exist, namely utilization of frequency counter connected via GPIB or USB to PC, realization of suitable frequency-to-voltage converter and voltage measurement using standard A/D converters, or direct estimation of frequency from output signal using fast A/D converter.

The third method is used in our measurement setup (see Fig. 4). A data-acquisition card (NI 5124) triggers on the rise-edges of the output signal and stores these corresponding times on which the instantaneous frequency is estimated by own-written software. Thus, our approach can acquire immediate change of QCM frequency in comparison to other measurement setups. Considering the knowledge of instantaneous frequency, chemical processes can be even described on the basis of high-order statistics.

4. Results and Discussion

In the last decade it is generally possible to observe the growth of research and utilization of polymers for active layers of chemical sensors. The experimental study was carried out on a sensor without and with a sorption layer of polypyrrole. The basic element is AT-cut quartz crystal of resonance frequency 10 MHz. This material is suitable for construction of QCM humidity sensors [15]. The change of relative humidity (RH) from 0 % to 25 % causes a drop of resonant frequency f in the range 50–150 Hz, response time during this exposition of humid air was about 50 s. Responses of these sensors were totally reversible (time constant of response ~ 30 s and time constant of recovery ~ 60 s). Thicknesses of sorption layers of QCM used to be in range 80–400 nm. Polypyrrole was deposited (in the cooperation with the Institute of Chemical Technology in Prague) by Matrix Assisted Pulsed Laser Evaporation, which seems to be perspective for preparation of sorption layers based on polymeric substances [16].

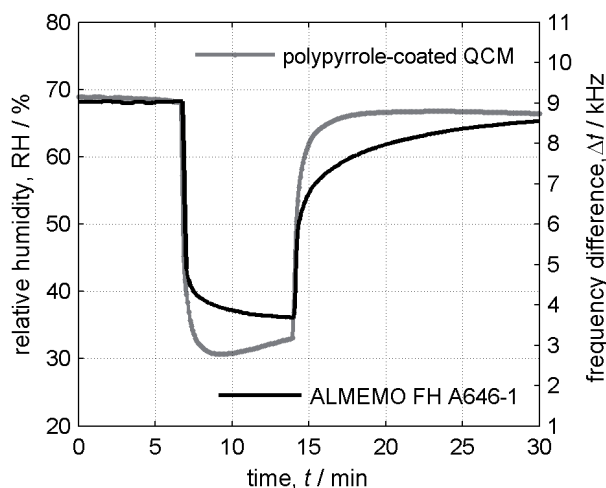


Fig. 5. Comparison of polypyrrole-coated QCM and a commercial humidity sensor during the controlled change of relative humidity.

Fig. 5 shows good agreement between relative humidity measurements, where commercial available humidity sensor (FH A646-1, ALBHORN) and QCM with deposited polypyrrole were used. As we expected, the commercial sensor has a longer time constant than polypyrrole-coated QCM. Relative humidity (RH) decrease from 69% to 30% causes a drop of frequency difference Δf

between the measuring oscillator and reference oscillator from 9150 Hz to 2783 Hz.

The measurements of QCM frequency fluctuation is analyzed in time domain and frequency domain. The measurable quantity is fluctuation of QCM frequency which is denoted by Δf . FFT of this realization is function of frequency and this variable in frequency domain is denoted by f . The frequency difference Δf fluctuation of two crystals without active layers in time domain has Gaussian distribution. Fig. 6 presents the noise spectrum of the measurement system, which is determined on the basis of this measurement provided on two identical quartz crystals without active layer.

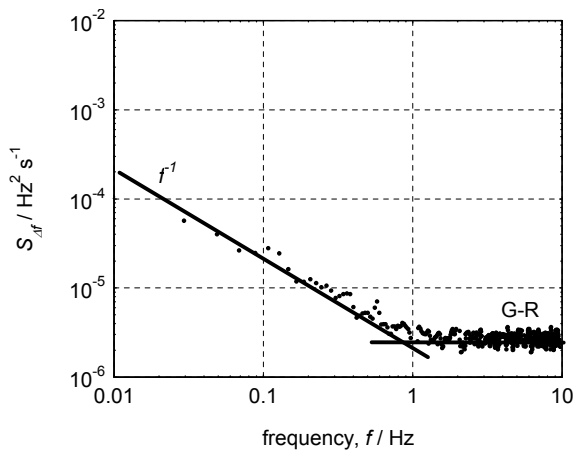


Fig. 6. Spectral density $S_{\Delta f}(f)$ of frequency difference fluctuation of the two identical quartz crystals without active layer.

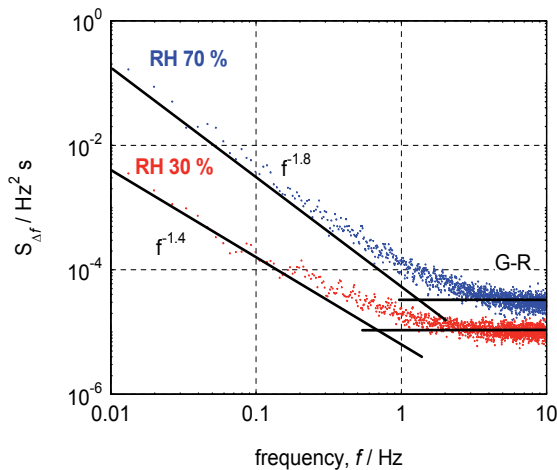


Fig. 7. Spectral density $S_{\Delta f}(f)$ of frequency difference fluctuations of the QCM sensor at relative humidity 30% and 70%.

The $1/f$ noise, thermal noise and generation-recombination (G-R) noise are the main noise components observed in QCM sensor as shown in Fig. 6 and Fig. 7. There are various models and mechanisms [17-20] that could be responsible for the observed noise but determination of these mechanisms is out of scope of the paper. Absorption of detected matter affects frequency fluctuations by two ways in dependence on a thickness of deposited sorbent

layer [6]. When the RH increases, the $1/f$ noise component changes insignificantly while the G-R component noticeably increases with a rise of RH value. It can be assumed that a shift of G-R noise is caused by increased flux density between the sorbent layer of QCM and the ambient environment. Considering this fact, we suggested a model of adsorption-desorption noise in QCM [6]. In the case of quartz crystal microbalance with thick sorbent layer (see Fig. 7), relative humidity increase represents shift of G-R component as well as $1/f$ noise component. We suppose that this change of $1/f$ noise component represents another generation-recombination process with long time constant. This process is probably caused by diffusion, which is connected to a potential barrier change due to ion adsorption of detected matter [6].

5. Conclusions

In our measurement setup, a quartz crystal oscillator with coated sorption layers and a reference quartz oscillator are driven by two oscillator circuits. Each one regulates frequency of one crystal at the minimum impedance which corresponds to series resonance. The mixing principle is used for frequency Δf measurements of these two independent oscillators. Signal to noise ratio is improved by low-pass (LP) filter after mixing procedure. It allows us separating of useful component of frequency Δf from both input signals and other parasitic components. A data-acquisition card triggers on the rise-edges of the output signal and stores these corresponding times on which the instantaneous frequency is estimated by own-written software. In comparison to other measurement setups, our approach can acquire immediate change of QCM frequency, thus, chemical processes can be even described on the basis of high-order statistics.

The experimental part of study was provided on quartz crystals without and with active layer, which is formed by deposited polypyrrole that is suitable for humidity sensor.

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Karel HAJEK was born in 1953. He received his M.Sc. degree in Electronics in 1976, and his Ph.D. in Electronics in 1981 from the Military Academy of Brno. From 2000 he is Professor at the University of Defense Brno in the field of Theoretical Electrical Engineering. His scientific activity is directed to the area of general circuit theory, especially to frequency filters, oscillators, low-noise amplifiers, optimization of the signal way with analog preprocessing and subsequent DSP. Further he is directed to application of signal processing nondestructive testing of materials, especially to special methods using nonlinear properties of defects.